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INVESTIGATION OF THE MAGNESIUM ANODE
SECOND QUARTERLY PROGRESS REPORT
1 OCTOBER 1962 TO 1 JANUARY 1963

SIGNAL CORPS CONTRACT NO. DA36-039-SC-89082
DEPARTMENT OF ARMY TASK NO. 3A99-09-001-02
U. S. ARMY ELECTRONICS RESEARCH AND DEVELOPMETN LABORATORY
FORT MONMOUTH, NEW JERSEY

THE DOW METAL PRODUCTS COMPANY
DIVISION OF THE DOW CHEMICAL COMPANY
MIDLAND, MICHIGAN

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INVESTIGATION OF THE MAGNESIUM ANODE

Report No. 2

SIGNAL CORPS CONTRACT NO. DA36-039-SC-89082

DEPARTMENT OF ARMY TASK NO. 3A99-09-001-02

SECOND QUARTERLY PROGRESS REPORT

1 OCTOBER 1962 TO 1 JANUARY 1963

OBJECT

The object of this work is the investigation of magnesium anode behaviors which affect primary cell application.

Prepared by: J. L. Robinson

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I. PURPOSE

The purpose of this research and development contract is the study of the electrochemistry involved in the use of magnesium as an anode in primary battery systems. The studies are a continuation of investigations initiated under Signal Corps Contract No. DA36-039-SC-88912.

II. ABSTRACT

Conflicting evidence was obtained as to whether or not the spontaneous corrosion of magnesium was enhanced by impressed anodic current. The apparent wasteful corrosion reaction of magnesium could be eliminated by increasing the electrode potential with magnesium chloride and perchlorate electrolytes. Low drain continuous service of magnesium dry cells was improved by the use of mixed acetate-perchlorate electrolytes.

III. CONFERENCES

On 28 November 1962, Mr. J. L. Robinson of The Dow Metal Products Company visited the U. S. Army Electronics Research and Development Laboratory, Fort Monmouth, New Jersey, to discuss the progress of this contract. Messrs. D. Wood, J. Murphy, J. Hovendon, and Dr. E. Baars represented the Signal Corps.

IV. DATA AND DISCUSSION

A. Anode Efficiency and Potential Behaviors

1. General Background

Three of the phenomena observed with magnesium anodes in aqueous environments are:

(a) The open circuit corrosion potential is more than a volt passive to the theoretical value for divalent ion formation. Further, the steady state working potential usually is insensitive to several hundred fold increases in applied anodic current.

(b) The apparent wasteful corrosion of a magnesium anode increases with increasing applied current. The end product of the corrosion reaction is hydrogen. This behavior is generally called the "Negative Difference Effect".

(c) Anodic potential transients are observed upon increasing or decreasing the current. The transient with increasing current results in the so called "delayed action" of magnesium dry cells.

There have been various explanations for the above phenomena. The view subscribed to by this laboratory is that all of these behaviors are the result of the protective magnesium hydroxide film on the electrode and processes which tend to damage or repair this film. On open circuit only a fraction of the electrode surface is active. The active area results from defects, undefined, in the magnesium hydroxide film. The measures working potential is a mixed potential, as described by Petrocelli,[1] of the anodic reaction $Mg \rightarrow Mg^{++} + 2e$ and the cathodic reaction $2H_2O + 2e \rightarrow H_2 + 2(OH)^-$. The potential of magnesium is sufficiently active to assume that the above cathodic reaction should occur directly on the exposed or active area of the electrode, as well as at discrete low overvoltage impurity sites. The magnesium hydroxide protective film tends to breakdown with increasing anodic current producing additional active area. The film breakdown results from build-up in concentration of a soluble magnesium salt at the interface which by buffering[2] increases the acidity at this site. The effective current density, and thereby the potential, changes very little over a wide range of anodic current because of the creation of the additional active area. Correspondingly, the increased area with increasing anodic drain results in additional surface for the cathodic reaction and hence results in the increase in the wasteful corrosion rate. The anodic voltage transients follow naturally since the establishment of steady film breakdown and repair rate is not instantaneous. One should be able to predict or reconcile specific anodic behaviors with the above general concepts if they are correct.

2. <u>Effect of Discharge Cycle On The Wasteful Corrosion Rate</u>

As measured by either weight loss or hydrogen evolution, the apparent wasteful corrosion rate associated with the continuous application of 50 milliamperes per square inch of anodic current is several orders of magnitude greater than the open circuit rate.

In accordance with the above behavior picture of film breakdown this higher corrosion rate should be maintained at the instant of removal of the applied current and should decrease with time towards the steady open circuit value as the film repairs. Such behavior is pictured in Figure 1. If this is correct, the total weight loss associated with the passage of a given number of coulombs should be larger if the current is passed intermittently rather than continuously. The increase in the weight loss should be a direct measure of the average corrosion rate during the open circuit portion of the discharge cycle. The shorter the open circuit period the greater the average open circuit corrosion rate, Figure 1. However, Kokoulina and Kabanov[3] found no apparent increase in the total hydrogen evolved from a magnesium anode when various values of current were passed intermittently rather than continuously. This observation would not be totally unexpected with the conditions of their experiments. They used current pulse frequencies ranging from 6 to 10,000 per second. The minimum pulse rate of six per second does not allow sufficient discharge time to build-up the magnesium ion concentration at the interface to the levels needed for the film breakdown process. This opinion is supported by the observation that 0.2 seconds or more are needed to complete the anodic voltage transient associated with an increase in current density[2].

The results of an investigation with AZ21X1 anodes and varying concentrations of $MgCl_2$, $Mg(Ac)_2$, and $Mg(ClO_4)_2$ electrolytes were shown in Report No. 1. The anodes were corroded at an anodic current density of approximately 50 milliamperes per square inch. The current was applied continuously and with various intermittent discharge cycles of equal on and off periods. The total discharge time was eight hours and the total test time was sixteen hours with the intermittent discharges. The average wasteful corrosion currents for the discharge and open circuit periods were calculated from measured weight losses. Report No. 1 gives details of the calculations. The experiment was repeated here since some of the original results were unexpected. The anode area was increased from the standard one square inch to three square inches for the repeat test, but the current density remained at 50 milliamperes per square inch. The data obtained were comparable to those of the previous test. As expected the apparent wasteful corrosion current decreased uniformly with increasing time for the open circuit period with $Mg(Ac)_2$ electrolytes, Table I and Figure 2. It was shown, Report No. 1, that the wasteful weight loss associated with the passage of a unit of current decreases slightly during the first 15 to 20 minutes of discharge. This would result in an error in the calculated corrosion currents which would tend to make the open circuit rates on the 5 and 10 minute cycles higher than the closed circuit rate as observed for some of the acetate electrolytes, Table I.

The data with the perchlorate electrolytes were so erratic that no attempt was made to analyze them, Table I. The data for the six normal concentration of this electrolyte can be completely ignored as the corrosion was intergranular and metal spalling occurred.

The data for the $MgCl_2$ electrolytes, while reasonably consistent and reproducible, are difficult to explain, Table I. There is a possible error, discussed below, which could move the apparent negative values of average corrosion rates to small positive values. However, low values for the corrosion rates with the short open circuit periods, even if positive, are not consistent with the visual observation that copious quantities of hydrogen are being evolved for an appreciable period after interruption of the circuit. The passage of the apparent corrosion rate through a maximum in the region of 15 to 20 minutes with increasing open circuit time is also puzzling. That the continuing hydrogen evolution on current interruption is the result of oxidation by water of a product of the primary anodic reaction can not be ruled out. Such a reaction product would probably have to form as a layer on the anode since the evidence is for appreciable life and little or no migration from the surface. However, it is difficult for us to believe that this hydrogen evolution is not the result of the spontaneous corrosion of magnesium enhanced by applied current because of film damage. In view of this it is proposed to carry out a few tests wherein the hydrogen evolution rates are directly measured. This will allow the establishment with greater certainty as to whether or not reasonable steady state conditions occur in the five and ten minute discharge periods.

An indication was obtained that the film on magnesium ages and becomes more protective with exposure time to $MgCl_2$ electrolyte. Two procedures were utilized to study the effect of discharge time on anode efficiency. First, the standard procedure of applying the current immediately after immersion of the anode in the electrolyte, measuring the weight losses after passage of various amounts of current, and calculating anode efficiencies. The second method differed only in that the anode was left on open circuit sixteen hours prior to applying the current. The average weight losses for the open circuit time were obtained by corrosion tests, Table II, and subtracted from the total weight losses prior to efficiency calculations. It is interesting to note that higher corrosion rates were obtained with a weak bright pickle surface, Table II. Irrespective of the anode state, the efficiency was significantly higher and affected less by discharge time when soaked the sixteen hours. The total weight loss was dominated by the open circuit weight loss for discharge times of ten minutes or less. Variation in this open circuit weight loss accounts for the scatter in the efficiency values. Acceleration of this film aging by intermittent discharge could partially account for the higher anode efficiencies measured with intermittent discharge as opposed to continuous discharge with $MgCl_2$ electrolytes, Table I.

3. Steady State Potential Behavior

Only potentiostatic measurements, as outlined in Report

No. 1, were employed for the subsequent anodic polarization curves. The reported potentials are referred to the saturated calomel electrode. In all tests the 0.03 centimeter diameter reference bridge tip was positioned initially 0.19 centimeters from the electrode face. Resistivities of the electrolytes at 70F are given in Report No. 1. Except where noted, the reported potentials were not corrected for the IR drop between the electrode and bridge tip since accurate values could not be estimated because of high currents and/or temperatures encountered. Copper coulometers were employed to obtain the average applied current but the current was also followed by recording the potential drop over a known resistance. Average currents calculated by the two methods agreed within one per cent. The total apparent anodic current was obtained by weight loss measurements. The apparent average corrosion current was obtained by subtracting the average applied current from the apparent total flow.

Complete data are listed in Tables IV to VIII for the following systems respectively: AZ21X1 anodes with 8 N $MgCl_2$, 6 N $Mg(ClO_4)_2$, 6 N $Mg(Ac)_2$, and 4 N $Mg(Ac)_2$ electrolytes, and pure magnesium anodes with 6 N $Mg(Ac)_2$ electrolyte. Figures 3 through 12 are curves showing the relationship between the potential and the average applied and corrosion currents for these systems.

In no case was there evidence of oxygen evolution at the anode even though the measured potential was increased (passive direction) to the region of five volts for all systems. Thus, it is obvious that the listed controlled potentials can be far removed from the actual potential of the electrode surface. However, it was also evident that changes in the controlled potential did effect a change in the electrode surface. The magnitudes of the applied and/or corrosion currents continued to depend upon the potential up to the five volts with the chloride and the perchlorate electrolytes, Figures 3, 5, and 6. Regions where the potential increased independently of the applied and corrosion currents were observed with acetate electrolytes, Figures 7 through 12, but marked progressive changes in the corrosion pattern occurred in these regions. Figure 13 shows the change in the corrosion from a highly uniform to a well developed cellular pattern as the potential was increased from -1.0 to +3.80 volts. The cellular structure was independent of the grain structure of the electrode. Changes in the corrosion pattern were also observed with chloride and perchlorate electrolytes. With these latter electrolytes the surface appearance changed from smooth but matte to smooth and bright (electropolish) to bright plus parallel grooves as the potential was increased. The grooves were not caused by gas errosion since they appeared after gas evolution ceased as discussed below. In all cases the changes in the corrosion patterns appeared to initiate at a potential where a maximum in the applied current was reached, Figures 3, 5, 7, 9, and 11.

In general, the observed relationships between potential and currents conformed to the anode behavior being governed by a protective film. To move the potential in the positive direction the anodic reaction has to be increased. This was done by applying current. Film damage and increased active area are associated with the increased anodic reaction in the fashion discussed above. The rate of the cathodic reaction, the wasteful corrosion reaction, per unit active area, should decrease with increasing potential. The initial increase in the total corrosion current with increasing potential indicates that the increase in the active area more than offsets any decrease in the intensity of the corrosion reaction. The corrosion current actually passed through a maximum and dropped to zero as expected with the chloride and perchlorate electrolytes, Figures 4 and 6. That the corrosion reaction actually ceased with these electrolytes was indicated by a lack of visible gas evolution. Measured anode efficiencies approaching 100% were actually obtained with the chloride electrolyte, Table IV. With the chloride electrolyte the visible gas evolution ceased after a short time at a measured potential of zero. This is not far removed from the potential region of a minus 0.3 to minus 0.7 volts where the cathode reaction might be expected to stop if this reaction were the reduction of water. The observation that corrosion current with 6 normal $Mg(Ac)_2$ reaches but does not pass through a maximum with increasing potential can not be explained at this time, Figures 8 and 12. There was an indication with the 4 N $Mg(Ac)_2$ electrolyte, Figure 10, that the same behavior as observed with chloride electrolyte might be obtained with lower $Mg(Ac)_2$ concentrations. This will be further explored with a two normal electrolyte.

With all systems, except perchlorate, there was a region where the potential changed independent of the applied current, Figures 3, 5, 7, 9, and 11. That these regions do not represent concentration polarization is indicated by the maxima in the current, Figures 3, 5, and 9, and the order of magnitude increases in the current obtained by changing anode composition with 6 N $Mg(Ac)_2$ electrolyte, Figures 7 and 11. This leaves as the likely cause, the formation at the anode surface of a film which is a potent barrier to the charge transfer step. If the film were stable it would be expected to grow and a corresponding decrease in current should be observed with time at a given potential. While there was very little evidence of such film growth, based on comparison of the final and average applied currents in the tables, such growth could have been masked by the increasing temperature of bulk electrolytes. (See final temperatures, Tables IV through VIII.) Further evidence of the lack of stability of the film is the recoveries of the potentials from the plus five volts to active values on removal of the applied current. A relatively slow recovery with a break in the curve at the potential at which the film first forms might be expected with a reasonably stable film. With AZ21X1 anodes and the chloride and the acetate electrolytes, the potential recovered in less than 0.02 seconds from a plus five volts to the original open circuit potential value. In less than 0.1 second the potential reached a value of a minus 1.80 volts which was considerably more active than the original open circuit value, Figure 14. There was no sign of breaks in the in the curves. Several minutes were required for the potentials to increase from the minus 1.80 volts to the region of the original open circuit value. This is substantially the behavior observed in all systems upon removal of an applied anodic current from magnesium when the potential is in the active region[2].

A much different behavior was observed with AZ21X1 anodes with the perchlorate electrolyte and pure magnesium anodes with the acetate electrolyte. With these systems the potential did not become more active than the original potential, but rather approached within 0.1 of a volt of the steady state value in less than 0.01 seconds after which it slowly approached the apparent rest potential in a cyclic fashion. This behavior is shown in Figure 15. No interpretation of the recovery curves has been made other than they do not represent behavior expected from a stable film.

The high currents encountered and the lack of temperature control of the bulk electrolyte has made it difficult to interpret the potentiostatic anodic polarization curves. The high currents rule out any estimate of the actual electrode surface potential. The increasing temperature throws doubt on any apparent relationship between the applied current and potential in regions where the potential appears to move independently. Much lower currents and better temperature control should be obtained if the tests were carried out at lower temperatures. This will be explored.

4. Transient Potential Behavior

Some work in the area of the transient behavior has been carried out. Some of the early results had to be discarded when it was discovered that the electrolyte solution being used had become contaminated. Recent results will be reported in the next report since there are not sufficient data to present any reasonably consistent picture. The major effort of the next quarter is planned for this area.

B. Dry Cell Data

It was previously reported, Report No. 1, that the performance of magnesium dry cells could be improved by using a mixed electrolyte of magnesium acetate and magnesium perchlorate. The improvement was mainly observed at a low drain, 180 ohms, continuous discharge of steel jacketed, "D" size cells. The total concentration of the electrolytes was three normal. Comparable results have now been obtained at total electrolyte concentrations of one, two, and four normals, Tables IX, X, and XI, respectively.

Only a limited number of dry cells are planned for future work. The purpose of these cells will be to evaluate salts of aromatic acids as electrolytes. In wet cells anode efficiencies in excess of 95% have been obtained with such electrolytes[4].

V. CONCLUSIONS

A firm conclusion as to whether or not the spontaneous corrosion of magnesium is enhanced by applied anodic current can not be made with the conflicting evidence at hand.

The anodic polarization of magnesium is at least partially film controlled.

The performance of magnesium dry cells at light continuous drains can be improved by the use of a mixed electrolyte of magnesium acetate and magnesium perchlorate.

VI. TENTATIVE PROGRAM, THIRD QUARTER

The main emphasis will be on the investigation of the anodic transient potential behavior.

Some hydrogen evolution measurements to determine steady state conditions with magnesium anodes operating in chloride electrolytes are planned.

A few low temperature anodic polarization curves will be determined.

VII. REFERENCES

1. J. V. Petrocelli, J. Electrochem Soc., 97, 10, 1950

2. J. L. Robinson, P. F. King, Ibid, 108, 36, 1961

3. D. V. Kokoulina, B. N. Kabanov, Russian J. Physical Chemistry, 34, 1165, 1960

4. Signal Corps Contract - DA36-039-SC88912

EFFECT OF DISCHARGE CYCLE ON PARASITIC CORROSION RATE

AZ21X1 ANODES, 3.2 SQ. INCHES, ~50 MA/IN^2, TOTAL CURRENT - 24 AMPERE MIN./IN^2

TEST CYCLE	% ANODE EFFICIENCY						APPARENT AVE. WASTEFUL CURRENT (Ica) MILLIAMPERES/IN^2						(1)
ON-MIN. →		5	10	15	20	30		5	10	15	20	30	3
OFF-MIN. →		5	10	15	20	30		5	10	15	20	30	DAYS
Mg (Ac)$_2$	CONTINUOUS						CLOSED CONTINUOUS	OPEN CIRCUIT					
1 NORMAL	88.1	76.9	78.5	79.3	80.4	81.9	6.9	8.3	6.9	6.2	5.4	4.3	.03
2 "	87.4	75.4	77.7	78.5	79.4	81.5	7.1	9.3	7.1	6.7	5.9	4.3	.04
4 "	87.0	74.6	76.5	77.1	78.2	80.0	7.4	9.6	9.0	7.5	6.6	5.1	1.91
6.2 "	83.3	77.3	77.8	78.4	78.4	79.1	10.0	4.7	4.5	4.2	3.6	3.0	2.53
Mg Cl$_2$													
1 NORMAL	62.6	64.5	62.4	62.4	61.0	62.0	30.0	-2.5	0.4	0.4	2.1	0.7	.19
2 "	62.4	64.0	62.2	62.4	61.8	62.3	30.4	-2.0	0.1	0	0.9	0.1	.84
4 "	61.2	62.4	60.2	60.3	59.3	60.7	31.8	-1.5	1.5	1.3	2.6	0.7	2.20
6 "	63.4	59.8	61.2	59.9	61.5	60.5	29.0	4.8	2.8	4.6	2.4	3.8	2.30
Mg (ClO$_4$)$_2$													
1 NORMAL	76.2	70.5	72.0	71.0	73.4	72.2	15.6	5.3	3.9	4.9	2.8	3.8	.03
2 "	70.3	67.5	70.9	68.5	71.7	69.6	21.1	3.0	-0.5	1.9	-1.3	0.9	.02
4 "	67.2	63.8	68.7	63.9	68.3	64.8	24.4	4.3	-1.5	4.0	-1.2	3.0	.01
6 "	44.0	53.0	58.5	47.3	52.0	44.7	63.6	-19.1	-28.1	-8.3	-17.4	-2.1	.10

(1) 3 DAY OPEN CIRCUIT STAGNANT IMMERSION TEST

TABLE I

16 HOUR STAGNANT IMMERSION CORROSION TEST
AZ21X1 ANODES, SIZE 2" x 2" x .146", ALLOY 95553

CONDITION [1]	ELECTROLYTE	WEIGHT LOSS-GRAMS	AVE.	MCD [3]	AVE.
EXTRUDED	2 N $MgCl_2$	.286		7.5	
		.289		7.6	
		.296		7.7	
		.281		7.3	
		.297	.286	7.5	7.5
[2] EXTRUDED + HEAT TREAT.	2 N $MgCl_2$	.154		4.0	
		.166		4.3	
		.147		3.9	
		.144		3.8	
		.138	.150	3.6	3.9
EXTRUDED + HEAT TREAT.	2 N $MgCl_2$ + 0.25 GMS/L Na_2CrO_4	.088		2.3	
		.082		2.2	
		.068		1.8	
		.088		2.3	
		.135	.094	3.5	2.4
EXTRUDED + HEAT TREAT + WEAK BRIGHT PICKLE	2 N $MgCl_2$ + 0.25 GMS/L Na_2CrO_4	.112		2.9	
		.110		2.9	
		.112		2.9	
		.098		2.6	
		.110	1.08	2.9	2.8

(1) ALL SAMPLES CAUSTIC CLEANED AND PICKLED ONE MIL PER SIDE IN ANP
(2) HEAT TREATMENT - 2 HOURS AT 900 F, H_2O QUENCHED
(3) MILLIGRAMS PER SQUARE CENTIMETER PER DAY

TABLE II

ANODE EFFICIENCY vs CONTINUOUS DISCHARGE TIME

AZ21X1 ANODES 2" x 2" x .146", ALLOY 99553

APPLIED CURRENT 400 MILLIAMPERES, 2 N $MgCl_2$ ELECTROLYTE

	CURRENT APPLIED IMMEDIATELY				ANODE PRE-SOAKED 16 HRS. IN ELECT.			
ANODE STATE ——	EXTRUDED	EXTRUDED + HEAT TREAT			EXTRUDED	EXTRUDED + HEAT TREAT		
SURFACE TREATMENT —	NONE	NONE	NONE	W.B.P.	NONE	NONE	NONE	W.B.P.
GMS/L Na_2CrO_4 ——	0	0	.25	.25	0	0	.25	.25
MINUTES DISCHARGED	% ANODE EFFICIENCY							
5	42	48	—	—	57	—	—	—
10	32	46	41	34	47	55	59	77
20	38	50	45	40	55	64	62	70
30	44	49	47	44	55	60	—	—
60	46	52	51	50	55	61	56	60
120	50	56	55	53	57	61	61	60
480	—	—	58	59	—	—	62	60

TABLE III

POTENTIOSTATIC DATA
AZ21X1 ANODES — 8 N $MgCl_2$

Mg ELECTRODE POTENTIAL [1] VOLTS vs SAT. CAL.	FINAL TEMP. °F	TEST TIME MINUTES	[2] CURRENTS MILLIAMPERES / IN^2					ANODE EFFICIENCY %
			I_{am}	I_{af}	I_a	I_{ca}	I_t	
+ 5.0	84	30	1720	860	726	12.2	738	98.3
+ 5.0	101	30	1560		818	8.4	836	99.0
+ 3.0	88	30	1210	810	655	11.0	676	98.5
+ 1.5	82	30	720	620	548	9.3	557	98.3
+ .75	81	30	750	680	589	12.4	601	97.9
0	80	30	800	650	710	34.8	735	95.5
− .50	—	30	1530	985	957	129	1086	88.2
− .75	102	30	2000	1850	1650	421	2071	79.4
− 1.0	84	20	2250	1800	1585	462	2047	77.5
− 1.25	93	30			1478	524	2002	74.0
− 1.40	84	30			1073	434	1507	71.4
− 1.50	80	30	700	630	652	288	940	69.4
− 1.55	79	30			474	217	691	68.6
− .65	89	30	1780	1080	1070	172	1242	86.3
− .70	92	30	1800	1550	1390	363	1553	79.4

TABLE IV

(1) NOT CORRECTED FOR IR DROP

(2) I_{am} = MAXIMUM APPLIED CURRENT
I_{af} = FINAL APPLIED CURRENT
I_a = AVERAGE APPLIED CURRENT
I_t = APPARENT TOTAL CURRENT FLOW FROM WT. LOSS
I_{ca} = APPARENT AVERAGE CORROSION CURRENT

POTENTIOSTATIC DATA – AZ21X1 ANODES – 6N $Mg(ClO_4)_2$

Mg ELECTRODE POTENTIAL [1] VOLTS vs SAT. CAL.	FINAL TEMP. °F	TEST TIME MINUTES	[2] CURRENTS MILLIAMPERES / IN^2					ANODE EFFICIENCY %
			I_{am}	I_{af}	I_a	I_{ca}	I_t	
+ 5.00 (4)	90	130	—	190	202	28.4	230.4	87.7
+ 4.00 (3)	81	130			306	51	357	85.8
+ 4.00 (4)	88	90	4,000	200	262	35	299	87.5
+ 3.50 (4)	92	38	5,400	730	598	109	707	84.5
+ 3.00 (3)	99	40	3,700	2,400	1,107	286	1,393	79.4
+ 2.50 (3)	90	17	3,630	2,640	1,765	493	2,258	78.3
+ 2.25 (4)	111	5	4,600	3,500	4,180	1,570	5,750	72.9
+ 2.00 (3)	117	6	10,560	7,040	9,110	3,388	12,498	72.9
+ 1.50 (3)	120	6	10,560	7,920	9,856	3,828	13,684	72.1
+ 1.00 (3)	115	6	9,245	8,180	8,860	3,225	12,085	72.2
0 (3)	123	7	10,500	7,820	9,180	3,485	12,663	72.7
– 0.90 (4)	98	10	3,450	3,300	3,105	1,350	4,455	69.7
– 1.00 (3)	90	25	2,980	1,830	2,344	1,019	3,363	69.8
– 1.25 (4)	84	10	1,400	1,310	1,295	736	2,031	63.7
– 1.35 (4)	79	20	—	530	477	413	890	53.6
– 1.40 (4)	73	10	72	—	38.5	7.9	46.2	82.9
– 1.50 (4)	72	2,880	11	7.5	7.0	11	65	65.0

(1) NOT CORRECTED FOR IR DROP

(2) I_{am} = MAXIMUM APPLIED CURRENT – I_{af} = FINAL APPLIED CURRENT – I_a = AVERAGE APPLIED CURRENT
I_t = APPARENT TOTAL CURRENT FLOW FROM WT. LOSS – I_{ca} = APPARENT AVERAGE CORROSION CURRENT

(3) ANODE AREA 3.2 CM^2

(4) ANODE AREA 6.8 CM^2

TABLE V

POTENTIOSTATIC DATA
AZ21X1 ANODES - 6 N $Mg(Ac)_2$ - 70°F

Mg ELECTRODE POTENTIAL (1) VOLTS vs SAT. CAL.	TEST TIME HOURS	(2) CURRENTS MILLIAMPERES / IN² I_{am}	I_{af}	I_a	I_{ca}	I_t	ANODE EFFICIENCY %
+ 3.80	6	550	75	96.7	31.3	128	75.6
+ 0.85	16	370	61	80.7	24.9	105.6	76.4
+ 0.85	6	390	94	110.5	33.7	144.2	76.7
− 0.20	6	400	100	103.2	31.6	133.8	77.2
− 0.40	6	155	101	102.9	31.1	134	76.8
− 0.55	6		104	109.1	32.9	142	76.8
− 0.65	6		105	102.5	33.0	135.5	75.7
− 0.80	6		98	99.9	31.9	131.8	75.7
− 0.90	6	175	103	112.6	32.5	145.6	77.6
− 0.95	6		103	111.3	30.7	142	78.5
− 1.00	6		110	113.8	30.2	144	79.0
− 1.20	6	128	107	111.6	28.0	139.6	79.9
− 1.35	6		74	72	15.5	89.3	82.7
− 1.45	6	48	41	36.8	7.3	44.1	83.5

(1) CORRECTED FOR IR DROP (RANGE 0.10 TO 0.20 VOLTS)
(2) I_{am} = MAXIMUM APPLIED CURRENT − I_{af} = FINAL APPLIED CURRENT
I_a = AVERAGE APPLIED CURRENT − I_t = APPARENT TOTAL CURRENT FLOW FROM WT. LOSS
I_{ca} = APPARENT AVERAGE CORROSION CURRENT

TABLE VI

POTENTIOSTATIC DATA
AZ21X1 ANODES — 4N $Mg(Ac)_2$

Mg ELECTRODE POTENTIAL [1]VOLTS vs SAT. CAL.	FINAL TEMP. °F	TEST TIME MINUTES	[2]CURRENTS MILLIAMPERES / IN^2 I_{am}	I_{af}	I_a	I_{ca}	I_t	ANODE EFFICIENCY %
+ 4.00	98	135	1200	275	278	89.5	367.5	75.7
+ 2.00	90	135	1350	310	282	90.0	372.0	75.9
+ 1.00	90	135	—	320	271	83.7	354.7	76.5
0	84	135	500	305	267	81.4	348.4	76.7
− 0.50	84	135	420	315	288	88.5	376.5	76.3
− 0.75	—	135	330	310	301	98.4	399.4	75.4
− 1.00	85	135	345	—	297	115.0	412.0	72.1
− 1.10	88	135	340	340	316	127.0	443.0	71.4
− 1.15	85	135	330	330	283	123.0	406.0	69.8
− 1.20	—	135	325	300	283	119.0	402.0	70.3
− 1.23	82	135	330	320	207	77.5	284.5	72.8
− 1.25	80	135	230	210	203	69.9	272.9	74.4
− 1.40	73	135	55	44	42.5	6.9	49.4	86.2

(1) NOT CORRECTED FOR IR DROP

(2) I_{am} = MAXIMUM APPLIED CURRENT − I_{af} = FINAL APPLIED CURRENT − I_a = AVERAGE APPLIED CURRENT
I_t = APPARENT TOTAL CURRENT FLOW FROM WT. LOSS − I_{ca} = APPARENT AVERAGE CORROSION CURRENT

TABLE VII

POTENTIOSTATIC DATA
PURE MAGNESIUM — 6N $Mg(Ac)_2$

Mg ELECTRODE POTENTIAL (1) VOLTS vs SAT. CAL.	FINAL TEMP. °F	TEST TIME MINUTES	(2) CURRENTS MILLIAMPERES / IN²					ANODE EFFICIENCY %
			I_{am}	I_{af}	I_a	I_{ca}	I_t	
+ 2.80 TO 3.00	160	20	2850	2850	1945	867	2812	69.2
+ 2.00	200	40	3160	2300	2295	794	3089	74.4
+ 1.60 " 2.00	144	31	1770	1770	1322	482	1804	73.4
+ 1.40 " 1.80	166	20	3200	3200	2075	748	2823	73.5
0	160	60	1740	1740	932	324	1256	74.3
− 0.50	97	155	385	335	354	131	485	73.2
− 1.00	82	360	145	130	135.5	41	176.5	76.8
− 1.25	80	420	90	85	81.5	18.2	99.7	81.8
− 1.35	74	420	78	70	69.8	13.2	83.0	84.2
− 1.38	73	420	68	66	59.3	10.9	70.2	84.5
− 1.40		420	57	53	49.6	9.5	59.1	84.0
− 1.50	72	420	37	33	32.5	7.6	40.1	81.0
− 1.50		420	35	31	30.2	7.8	38.0	79.5
− 1.55		1260	27	20	22.4	9.2	31.6	70.9

(1) NOT CORRECTED FOR IR DROP

(2) I_{am} = MAXIMUM APPLIED CURRENT — I_{af} = FINAL APPLIED CURRENT — I_a = AVERAGE APPLIED CURRENT
I_t = APPARENT TOTAL CURRENT FLOW FROM WT. LOSS — I_{ca} = APPARENT AVERAGE CORROSION CURRENT

TABLE VIII

MIXED ACETATE — PERCHLORATE ELECTROLYTES

TOTAL NORMALITY 1.0

"D" SIZE CELLS, CONTINUOUS DRAIN, 70°F

ELECTROLYTE			BATCH	10 OHMS TO 0.75 VOLT				50 OHMS TO 1.00 VOLT			
				VOLTAGES				VOLTAGES			
$Mg(Ac)_2$	$Mg(ClO_4)_2$	$\frac{(Ac)^-}{(ClO_4)^-}$	No.	INITIAL C.C.	AVERAGE C.C.	HOURS CAPACITY	ANODE EFF. %	INITIAL C.C.	AVERAGE C.C.	HOURS CAPACITY	ANODE EFF. %
1.0	0	—	235182	1.55	1.13	21.5	83	1.73	1.38	156	73
0.75	.25	3	235208	1.77	1.19	22	78	1.84	1.33	145	76
0.50	.50	1	235209	1.78	1.23	22	73	1.86	1.37	170	74
0.25	.75	.33	235210	1.82	1.26	23	70	1.86	1.37	170	74
0	1.0	—	235211	1.88	1.38	24	74	1.92	1.40	175	65
				100 OHMS TO 1.10 VOLTS				180 OHMS TO 1.20 VOLTS			
1.0	0		235182	1.78	1.44	265	63	1.83	1.47	510	57
0.75	.25		235208	1.84	1.42	335	70	1.84	1.48	520	61
0.50	.50		235209	1.86	1.42	370	66	1.87	1.46	630	63
0.25	.75		235210	1.86	1.44	365	67	1.87	1.50	615	62
0	1.0		235211	1.92	1.50	310	53	1.95	1.54	490	53

TABLE IX

MIXED ACETATE — PERCHLORATE ELECTROLYTES

TOTAL NORMALITY 2.0

"D" SIZE CELLS, CONTINUOUS DRAIN, 70° FAHR.

ELECTROLYTE			BATCH	10 OHMS TO 0.75 VOLT				50 OHMS TO 1.00 VOLT			
				VOLTAGES				VOLTAGES			
$Mg(Ac)_2$	$Mg(ClO_4)_2$	$\frac{(Ac)^-}{(ClO_4)^-}$	No.	INITIAL C.C.	AVERAGE C.C.	HOURS CAPACITY	ANODE EFF.	INITIAL C.C.	AVERAGE C.C.	HOURS CAPACITY	ANODE EFF.
2.0	0	—	235190	1.71	1.13	30	88	1.81	1.37	171	71
1.5	0.5	3	235205	1.79	1.16	26	78	1.86	1.35	185	72
1.0	1.0	1	235206	1.84	1.22	25	76	1.89	1.38	176	72
0.5	1.5	.3	235207	1.88	1.28	26	70	1.92	1.38	170	76
0	2.0	—	235196	1.93	1.33	31	72	1.98	1.45	177	68
				100 OHMS TO 1.10 VOLTS				180 OHMS TO 1.20 VOLTS			
2.0	0		235190	1.80	1.45	315	72	1.82	1.50	< 480	50
1.5	0.5		235205	1.85	1.43	380	71	1.87	1.50	650	70
1.0	1.0		235206	1.92	1.43	350	67	1.92	1.49	630	68
0.5	1.5		235207	1.93	1.46	350	65	1.94	1.50	580	63
0	2.0		235196	1.93	1.51	290	52	2.01	1.59	475	53

TABLE X

MIXED ACETATE — PERCHLORATE ELECTROLYTES

TOTAL NORMALITY 4.0

"D" SIZE CELLS, CONTINUOUS DRAIN, 70° F

ELECTROLYTE			BATCH	10 OHMS TO 0.75 VOLT				50 OHMS TO 1.00 VOLT			
				VOLTAGES				VOLTAGES			
$Mg(Ac)_2$	$Mg(ClO_4)_2$	$\frac{(Ac)^-}{(ClO_4)^-}$	No.	INITIAL C.C.	AVERAGE C.C.	HOURS CAPACITY	ANODE EFF. %	INITIAL C.C.	AVERAGE C.C.	HOURS CAPACITY	ANODE EFF. %
4.0	0		235185	1.71	0.96	25	85	1.86	1.35	169	79
3.5	0.5	7	235214	1.78	1.05	26	84	1.86	1.28	155	72
3.0	1.0	3	235197	1.80	1.07	28	84	1.86	1.35	>141	72
2.0	2.0	1	235195	1.87	1.16	27	73	1.93	1.38	171	66
1.0	3.0	.3	235204	1.90	1.23	28	68	1.96	1.40	178	66
0.5	3.5	.14	235215	1.93	1.22	31	72	1.96	1.46	171	70
0	4.0		235202	1.94	1.37	28	72	1.99	1.47	161	69
				100 OHMS TO 1.10 VOLTS				180 OHMS TO 1.20 VOLTS			
4.0	0		235185	1.84	1.39	375	72	1.93	1.50	465	53
3.5	0.5		235214	1.90	1.43	305	68	1.87	1.50	600	62
3.0	1.0		235197	1.85	1.42	335	69	1.90	1.49	635	64
2.0	2.0		235195	1.95	1.41	355	61	1.95	1.46	605	64
1.0	3.0		235204	1.98	1.50	300	65	1.97	1.53	540	64
0.5	3.5		235215	1.90	1.51	360	68	1.94	1.55	600	64
0	4.0		235202	1.96	1.57	315	64	2.01	1.62	485	59

TABLE XI

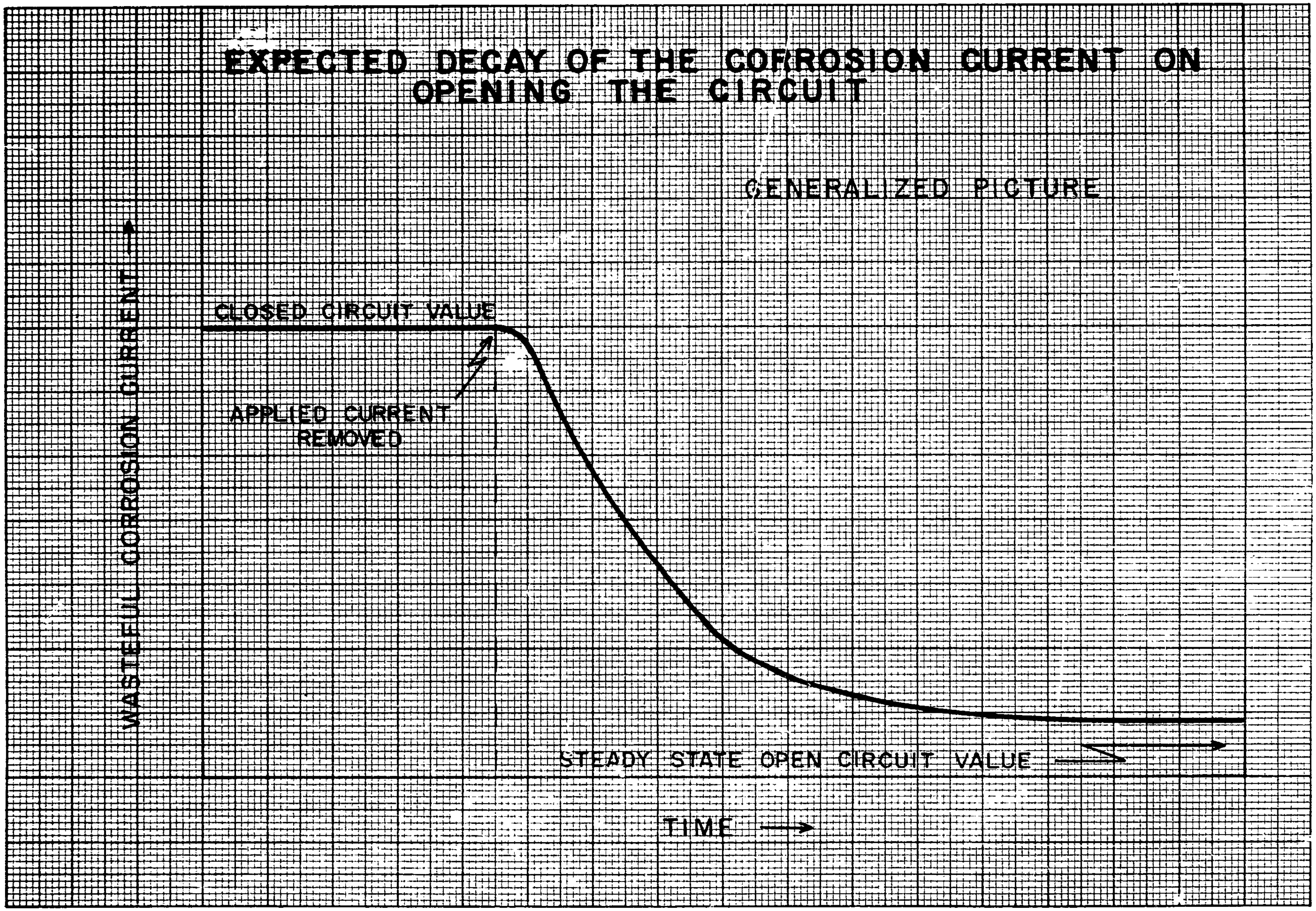
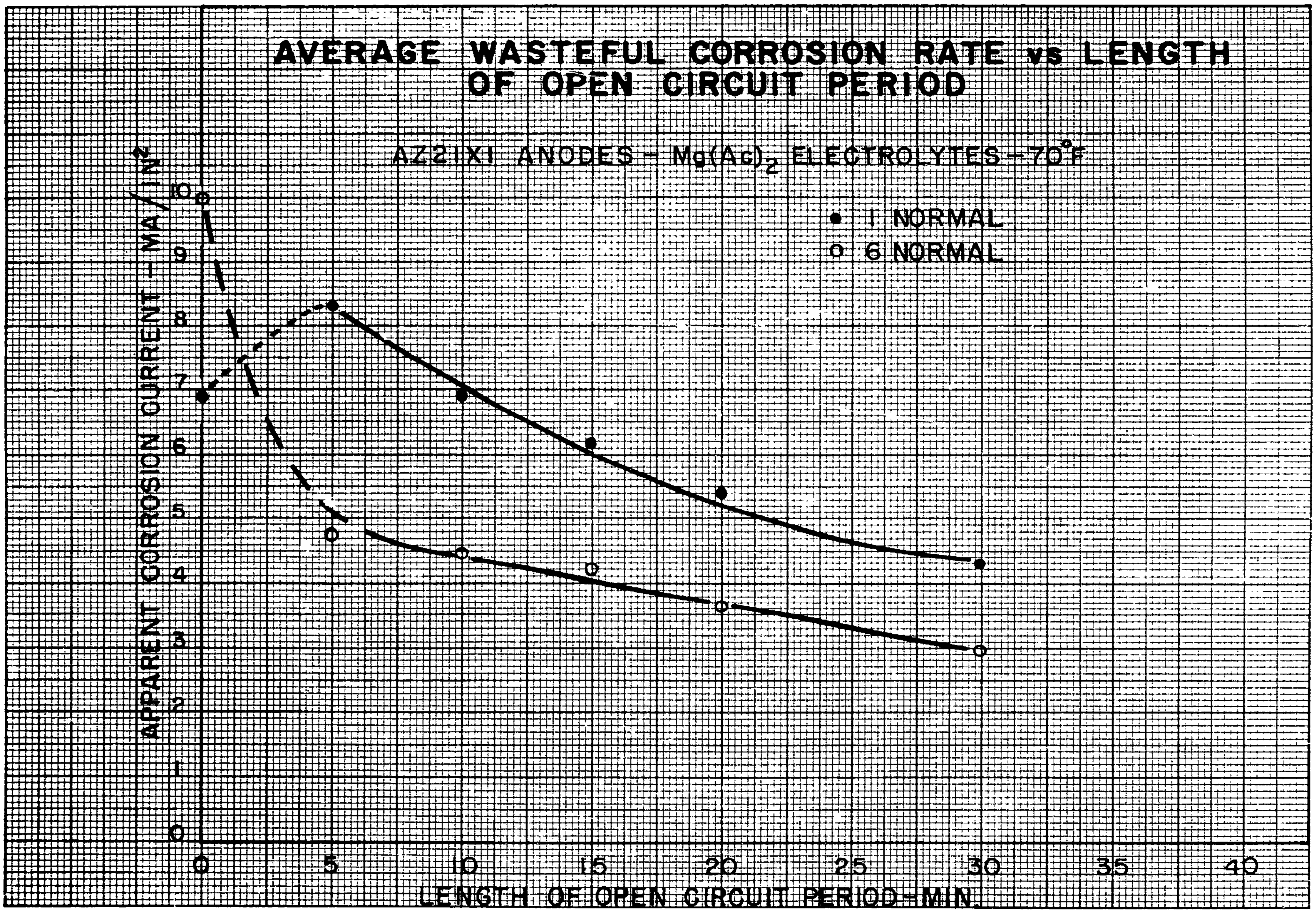
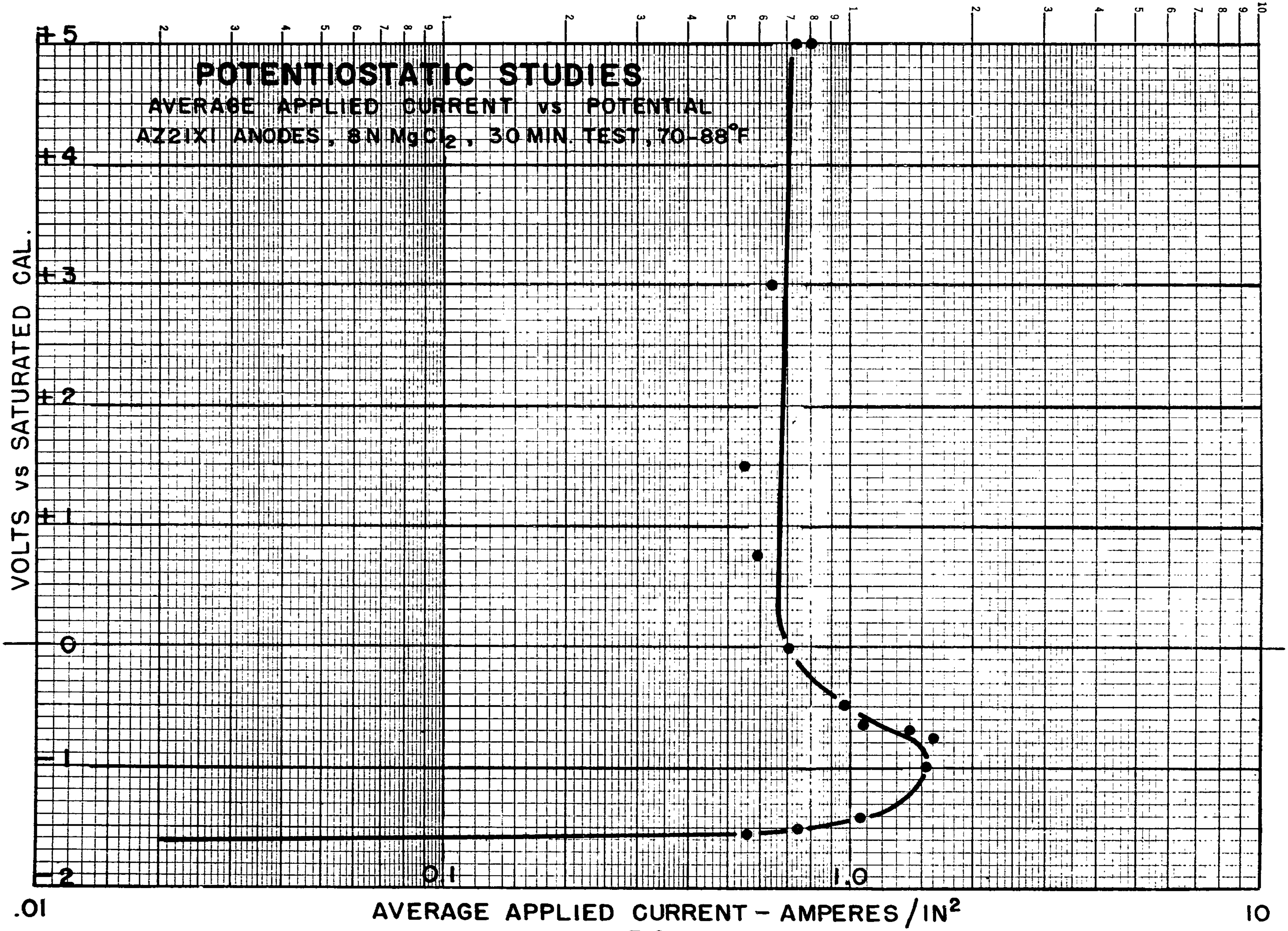

EXPECTED DECAY OF THE CORROSION CURRENT ON OPENING THE CIRCUIT
GENERALIZED PICTURE
WASTEFUL CORROSION CURRENT →
CLOSED CIRCUIT VALUE
APPLIED CURRENT REMOVED
STEADY STATE OPEN CIRCUIT VALUE
TIME →

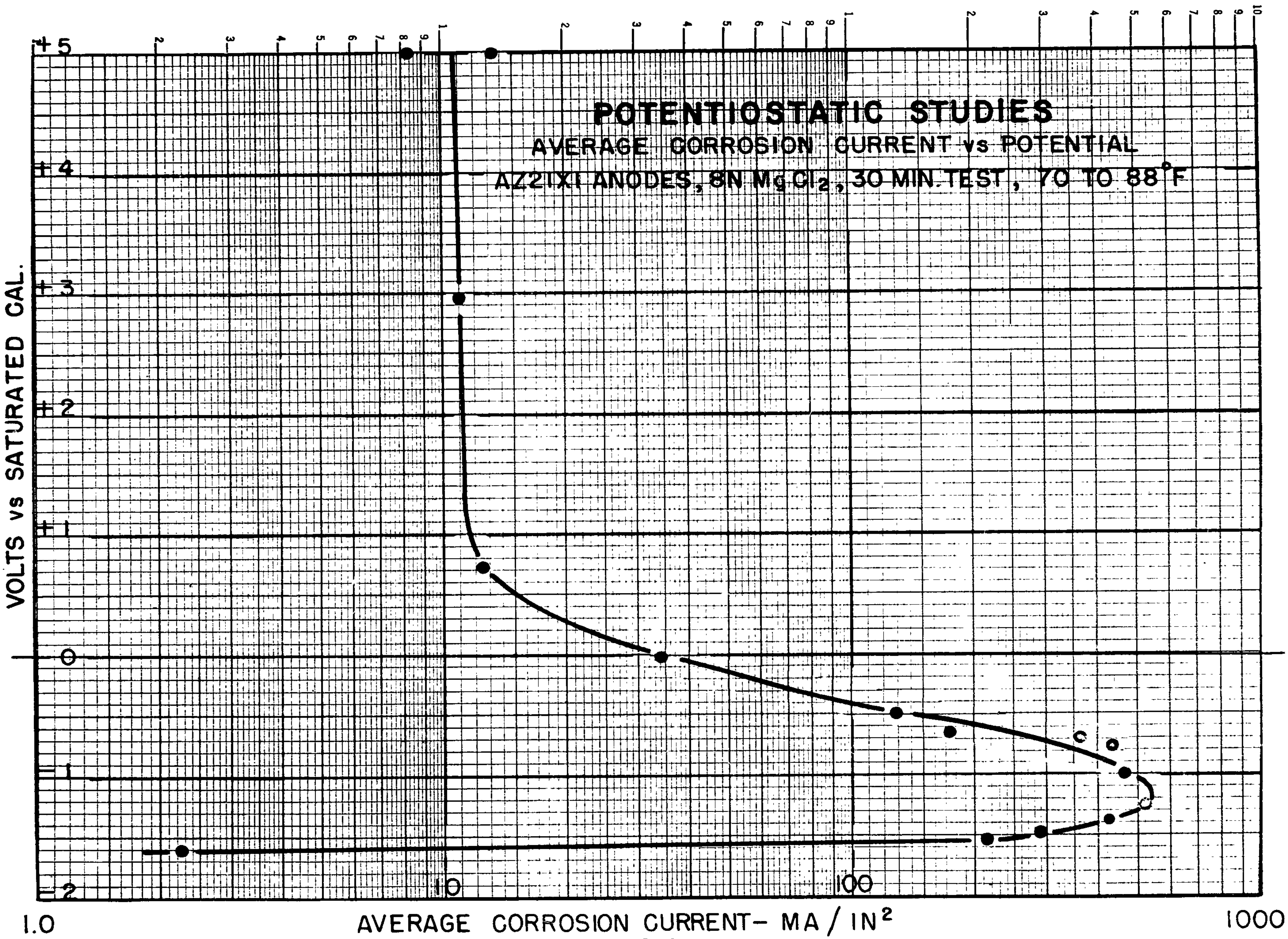
FIG. I



AVERAGE WASTEFUL CORROSION RATE vs LENGTH OF OPEN CIRCUIT PERIOD
AZ21XI ANODES - $Mg(Ac)_2$ ELECTROLYTES - 70°F
• 1 NORMAL
o 6 NORMAL
APPARENT CORROSION CURRENT - MA/IN²
LENGTH OF OPEN CIRCUIT PERIOD - MIN.

FIG. 2


POTENTIOSTATIC STUDIES
AVERAGE APPLIED CURRENT vs POTENTIAL
AZ21XI ANODES, 8N MgCl2, 30 MIN. TEST, 70-88°F
VOLTS vs SATURATED CAL.
+5
+4
+3
+2
+1
0
-1
-2
.01
0.1
1.0
10
AVERAGE APPLIED CURRENT - AMPERES / IN2
FIG. 3


POTENTIOSTATIC STUDIES
AVERAGE CORROSION CURRENT vs POTENTIAL
AZ21X1 ANODES, 8N $MgCl_2$, 30 MIN. TEST, 70 TO 88°F
VOLTS vs SATURATED CAL.
+5
+4
+3
+2
+1
0
-1
-2
1.0
10
100
1000
AVERAGE CORROSION CURRENT– MA / IN²
FIG. 4

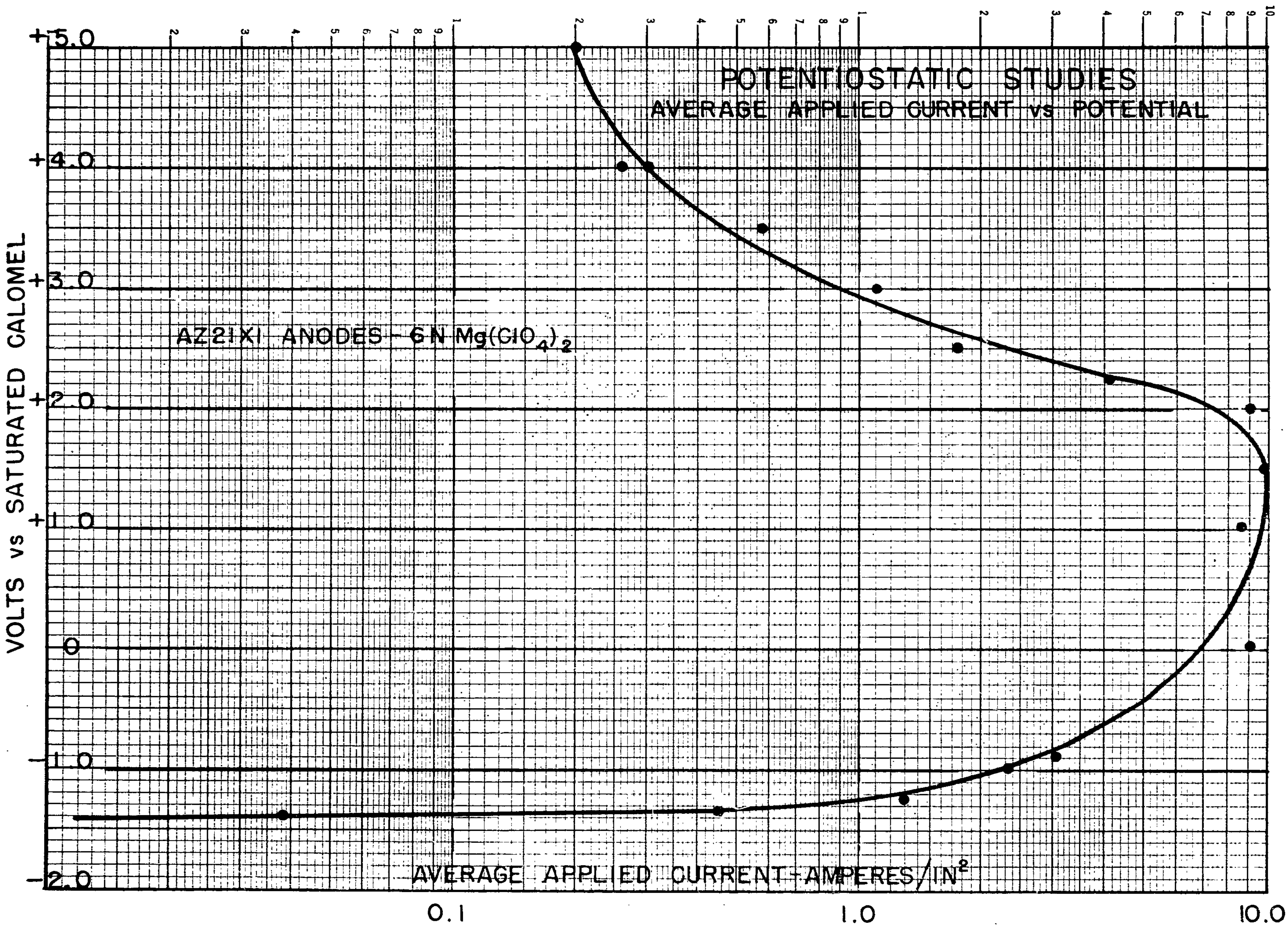

POTENTIOSTATIC STUDIES
AVERAGE APPLIED CURRENT vs POTENTIAL
AZ21XI ANODES - 6N $Mg(ClO_4)_2$
VOLTS vs SATURATED CALOMEL
+5.0
+4.0
+3.0
+2.0
+1.0
0
-1.0
-2.0
AVERAGE APPLIED CURRENT - AMPERES/IN²
0.1
1.0
10.0

FIG. 5

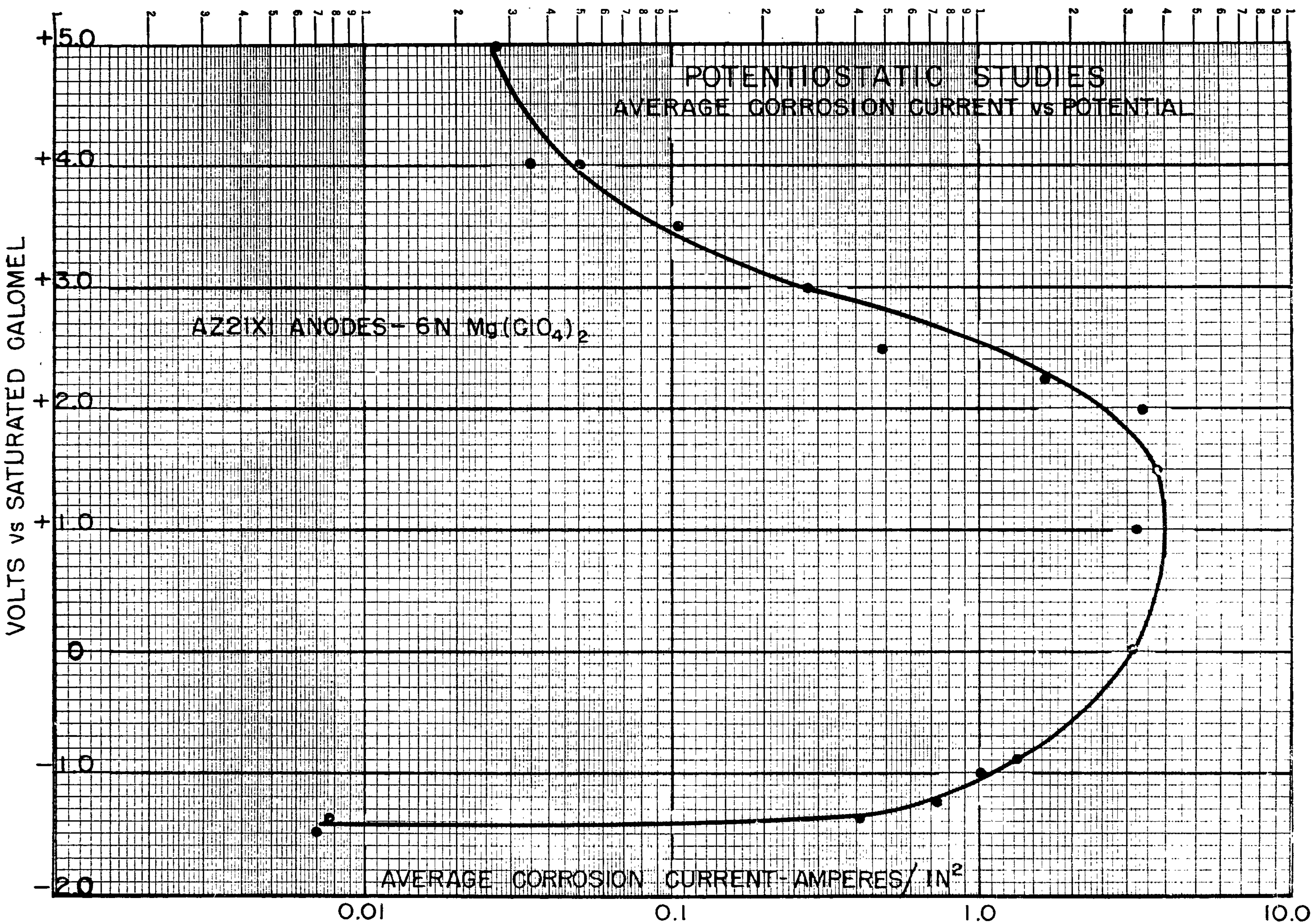

POTENTIOSTATIC STUDIES
AVERAGE CORROSION CURRENT vs POTENTIAL
AZ21XI ANODES - 6N Mg(ClO4)2
VOLTS vs SATURATED CALOMEL
+5.0
+4.0
+3.0
+2.0
+1.0
0
-1.0
-2.0
AVERAGE CORROSION CURRENT-AMPERES/IN²
0.01
0.1
1.0
10.0

FIG. 6

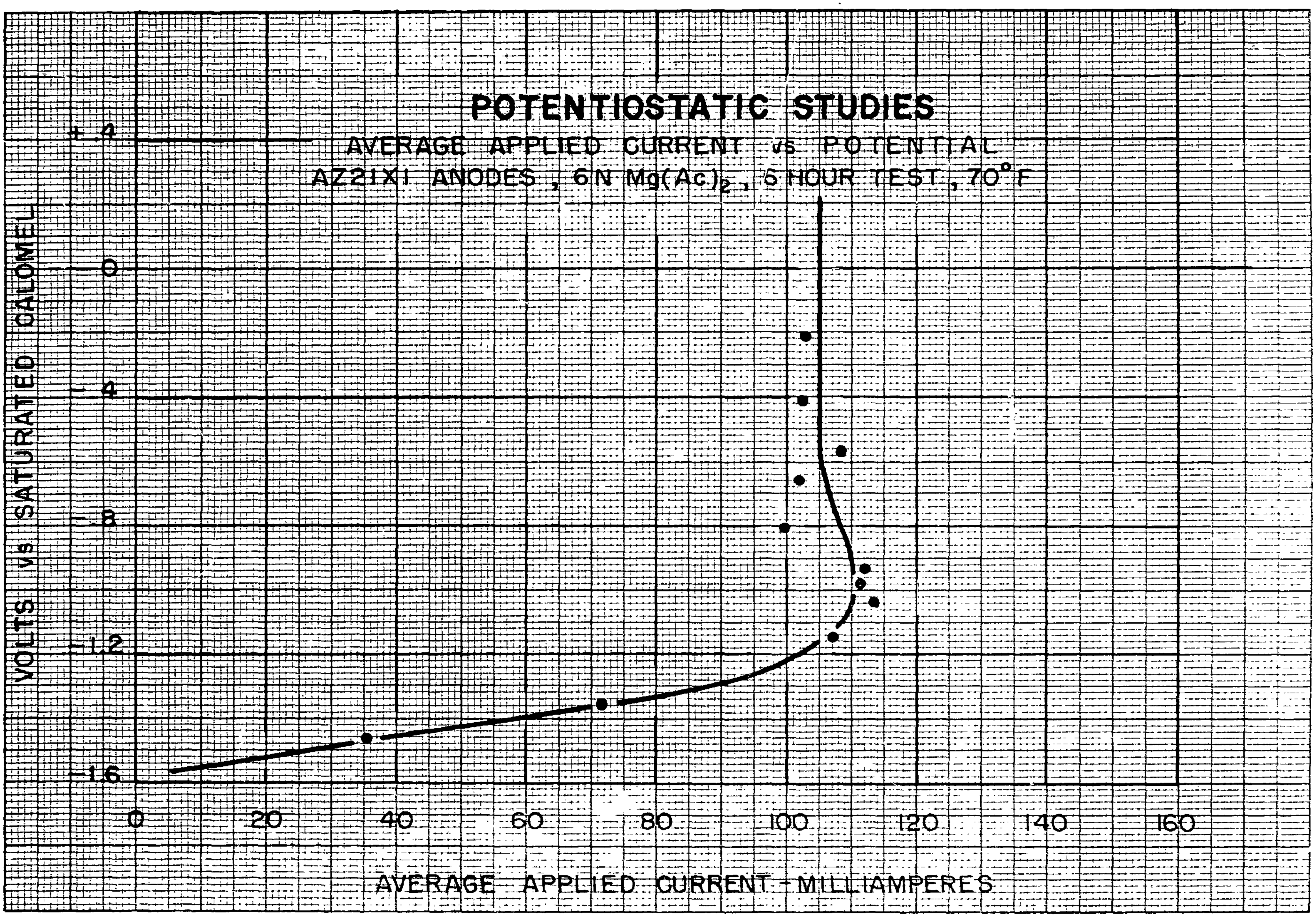

POTENTIOSTATIC STUDIES
AVERAGE APPLIED CURRENT vs POTENTIAL
AZ21X1 ANODES, 6N Mg(Ac)2, 6 HOUR TEST, 70°F
VOLTS vs SATURATED CALOMEL
+.4
0
-.4
-.8
-1.2
-1.6
0
20
40
60
80
100
120
140
160
AVERAGE APPLIED CURRENT - MILLIAMPERES

FIG. 7

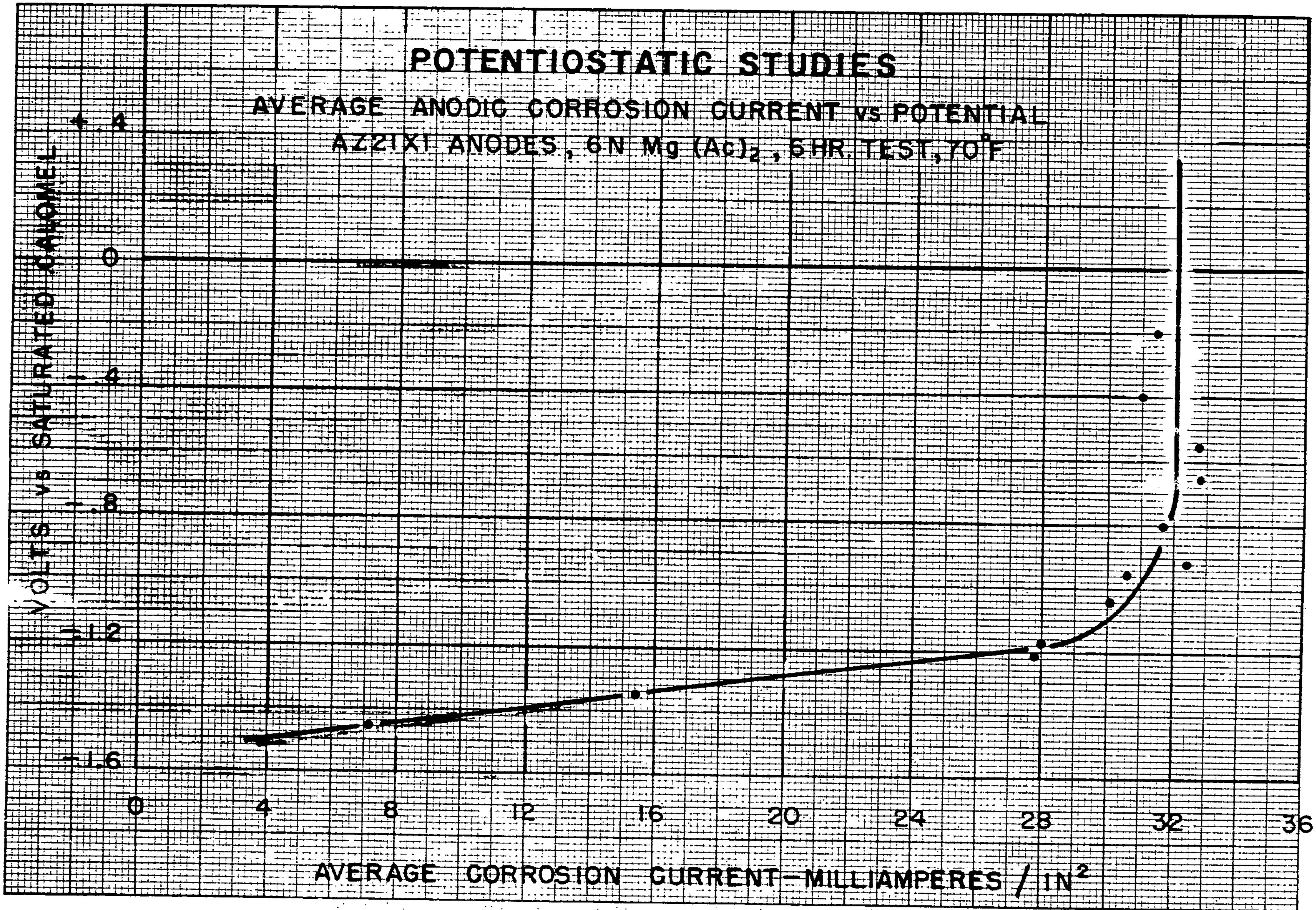

POTENTIOSTATIC STUDIES
AVERAGE ANODIC CORROSION CURRENT vs POTENTIAL
AZ21X1 ANODES, 6N Mg (Ac)2, 6 HR. TEST, 70°F
VOLTS vs SATURATED CALOMEL
+.4
0
-.4
-.8
-1.2
-1.6
0
4
8
12
16
20
24
28
32
36
AVERAGE CORROSION CURRENT—MILLIAMPERES / IN²

FIG. 8

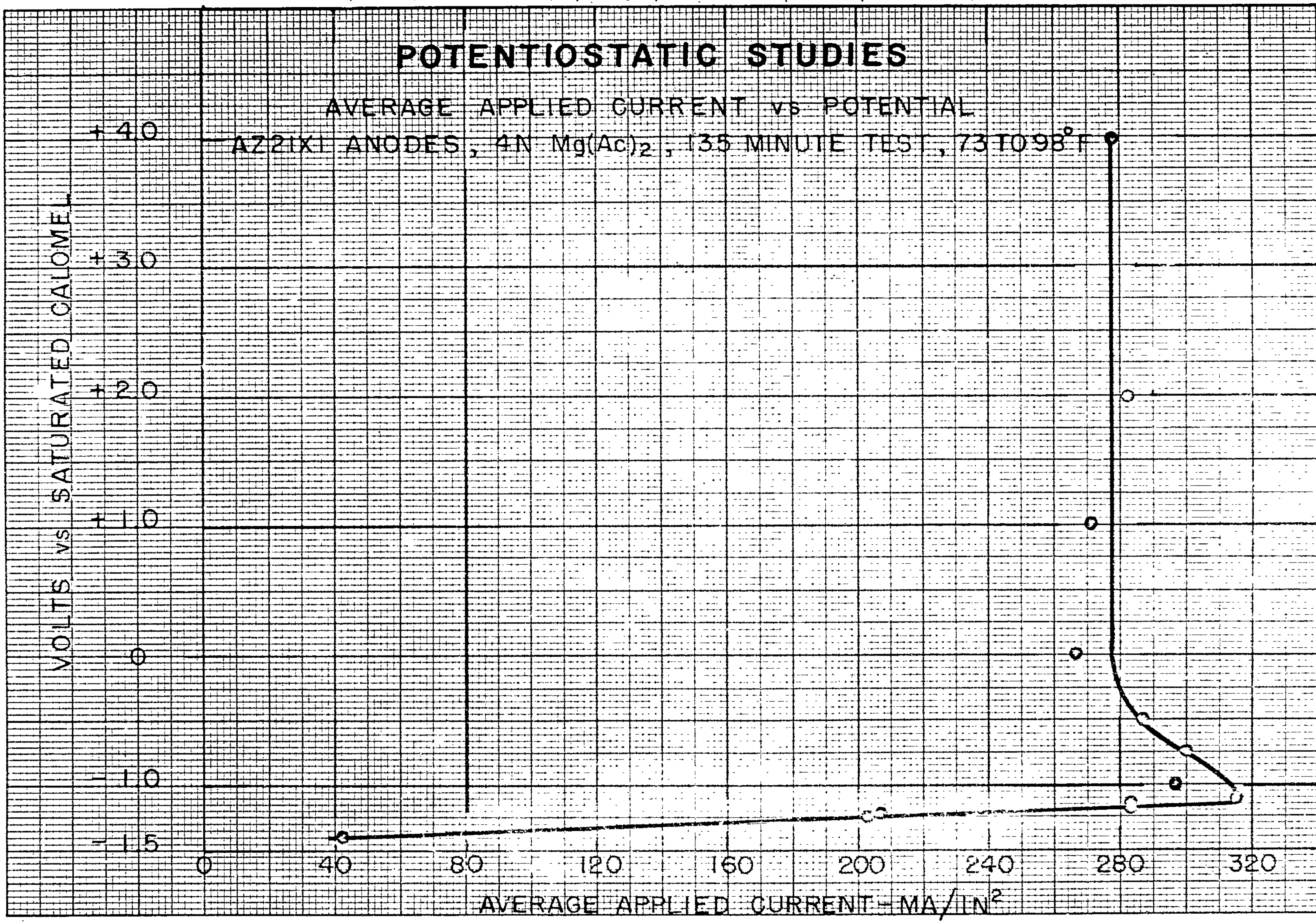

POTENTIOSTATIC STUDIES
AVERAGE APPLIED CURRENT vs POTENTIAL
AZ21X1 ANODES, 4N $Mg(Ac)_2$, 135 MINUTE TEST, 73 TO 98°F
VOLTS vs SATURATED CALOMEL
+4.0
+3.0
+2.0
+1.0
0
-1.0
-1.5
0
40
80
120
160
200
240
280
320
AVERAGE APPLIED CURRENT - MA/IN^2

FIG. 9

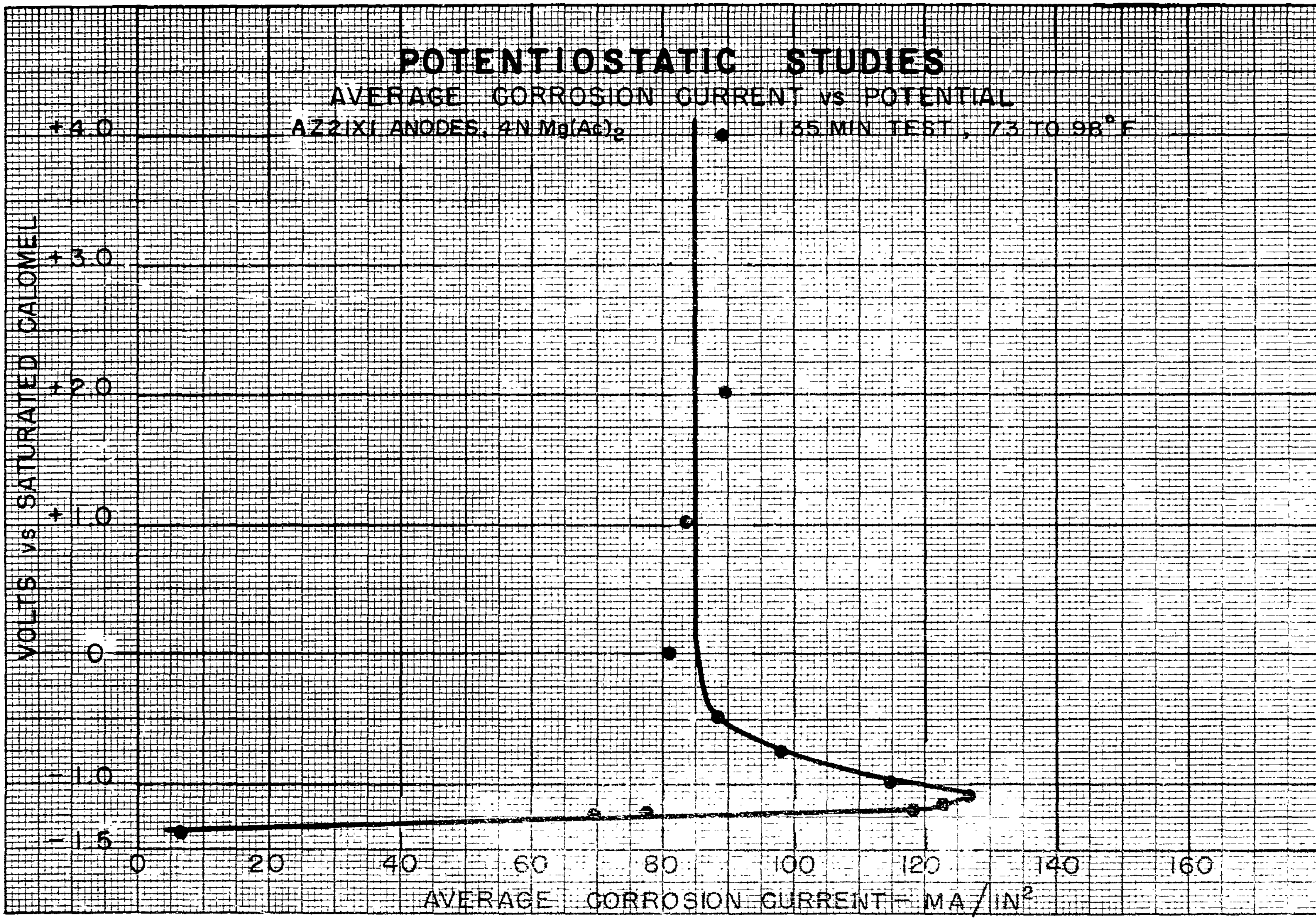

POTENTIOSTATIC STUDIES
AVERAGE CORROSION CURRENT vs POTENTIAL
AZ21XI ANODES, 4N Mg(Ac)2
135 MIN TEST, 73 TO 98° F
VOLTS vs SATURATED CALOMEL
+4.0
+3.0
+2.0
+1.0
0
-1.0
-1.5
0
20
40
60
80
100
120
140
160
AVERAGE CORROSION CURRENT - MA / IN²

FIG. 10

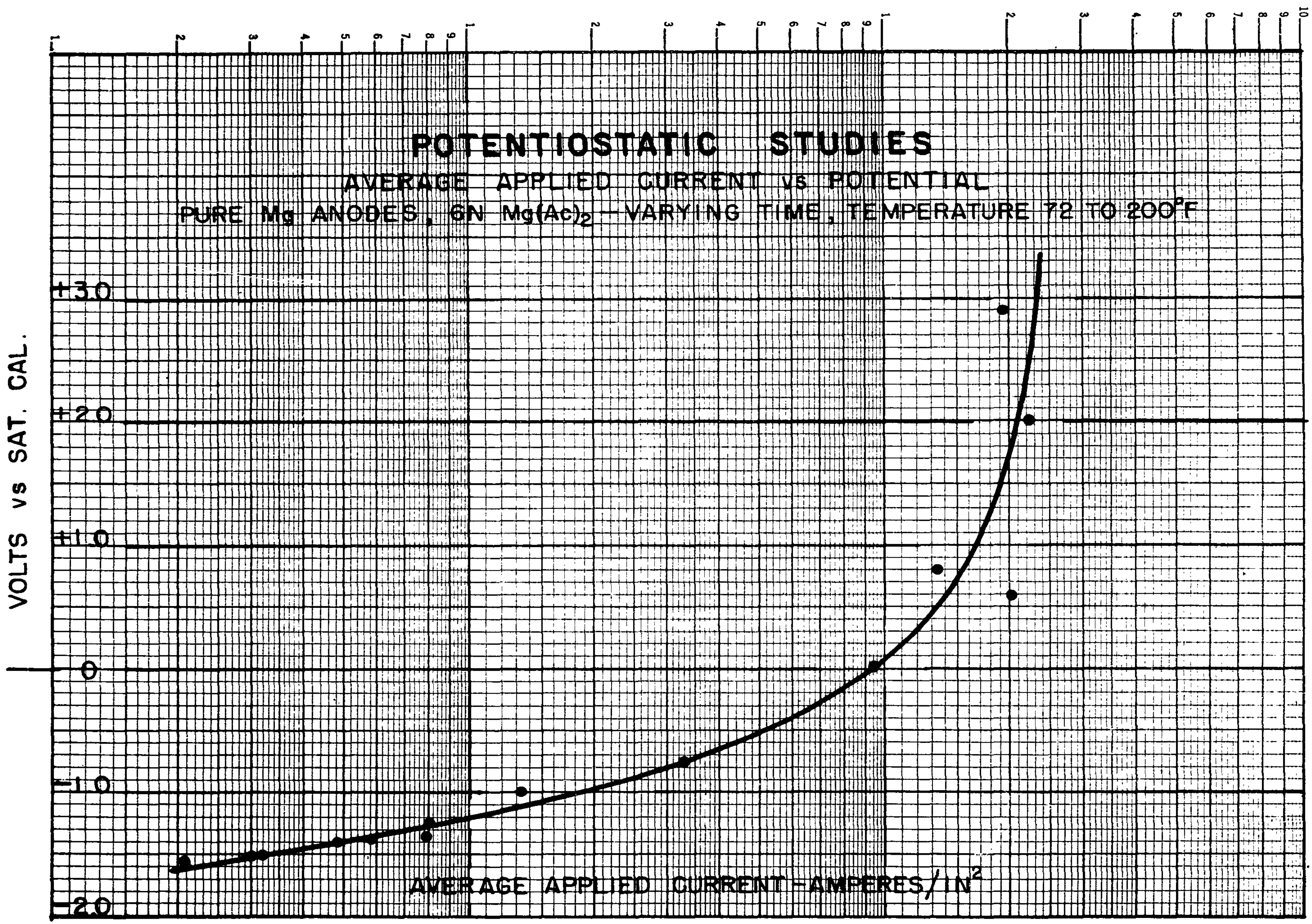

POTENTIOSTATIC STUDIES
AVERAGE APPLIED CURRENT vs POTENTIAL
PURE Mg ANODES, 6N Mg(Ac)2 – VARYING TIME, TEMPERATURE 72 TO 200°F
VOLTS vs SAT. CAL.
+3.0
+2.0
+1.0
0
–1.0
–2.0
AVERAGE APPLIED CURRENT – AMPERES/IN2
0.1
1.0
10

FIG. 11

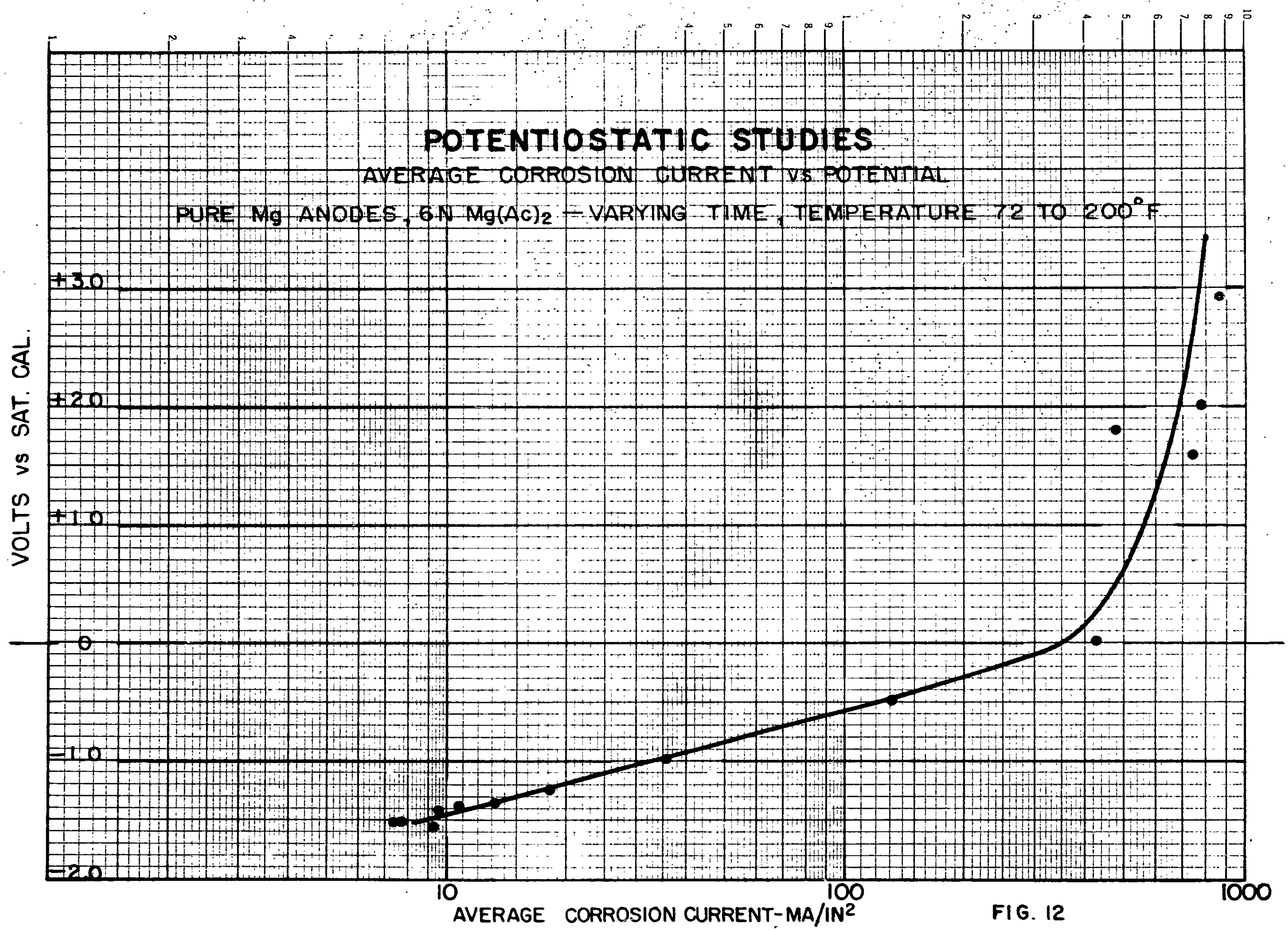
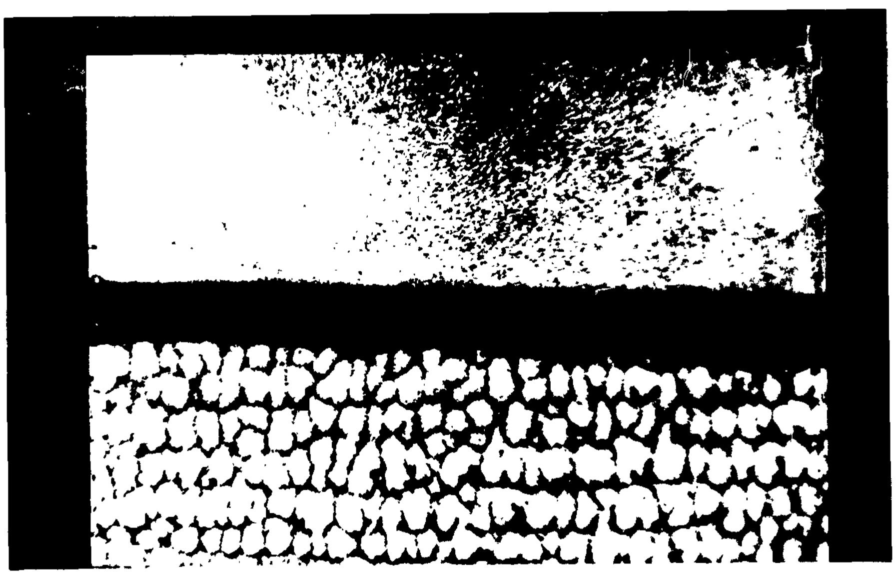

POTENTIOSTATIC STUDIES
AVERAGE CORROSION CURRENT vs POTENTIAL
PURE Mg ANODES, 6N Mg(Ac)2 — VARYING TIME, TEMPERATURE 72 TO 200°F
VOLTS vs SAT. CAL.
+3.0
+2.0
+1.0
0
-1.0
-2.0
10
100
1000
AVERAGE CORROSION CURRENT-MA/IN2

FIG. 12

ANODIC CORROSION PATTERN

AZ21X1 ANODES - 6N $Mg(Ac)_2$ ELECTROLYTE

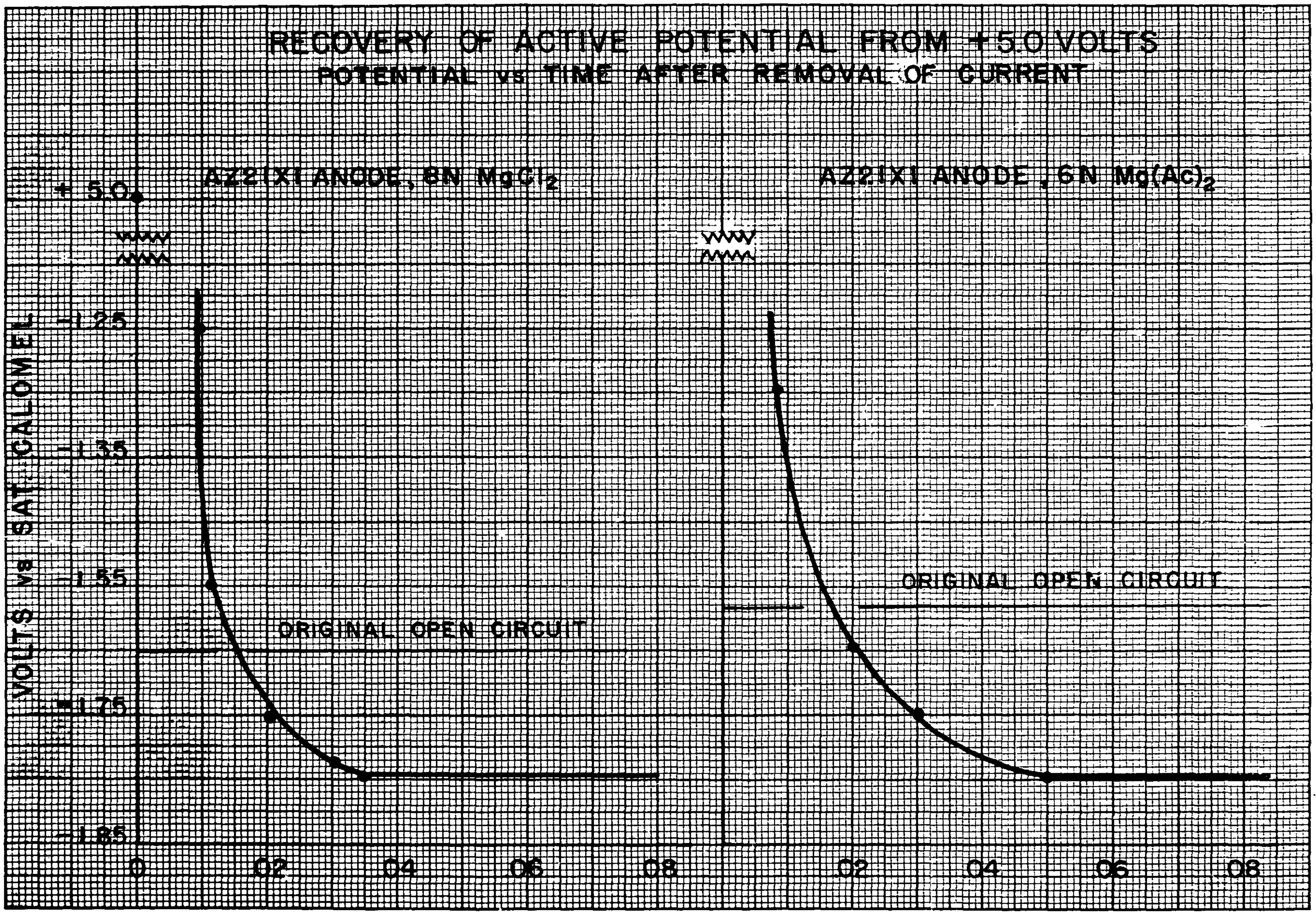


ANODE POTENTIAL
- 1.0 VOLT

+ 3.8 VOLTS

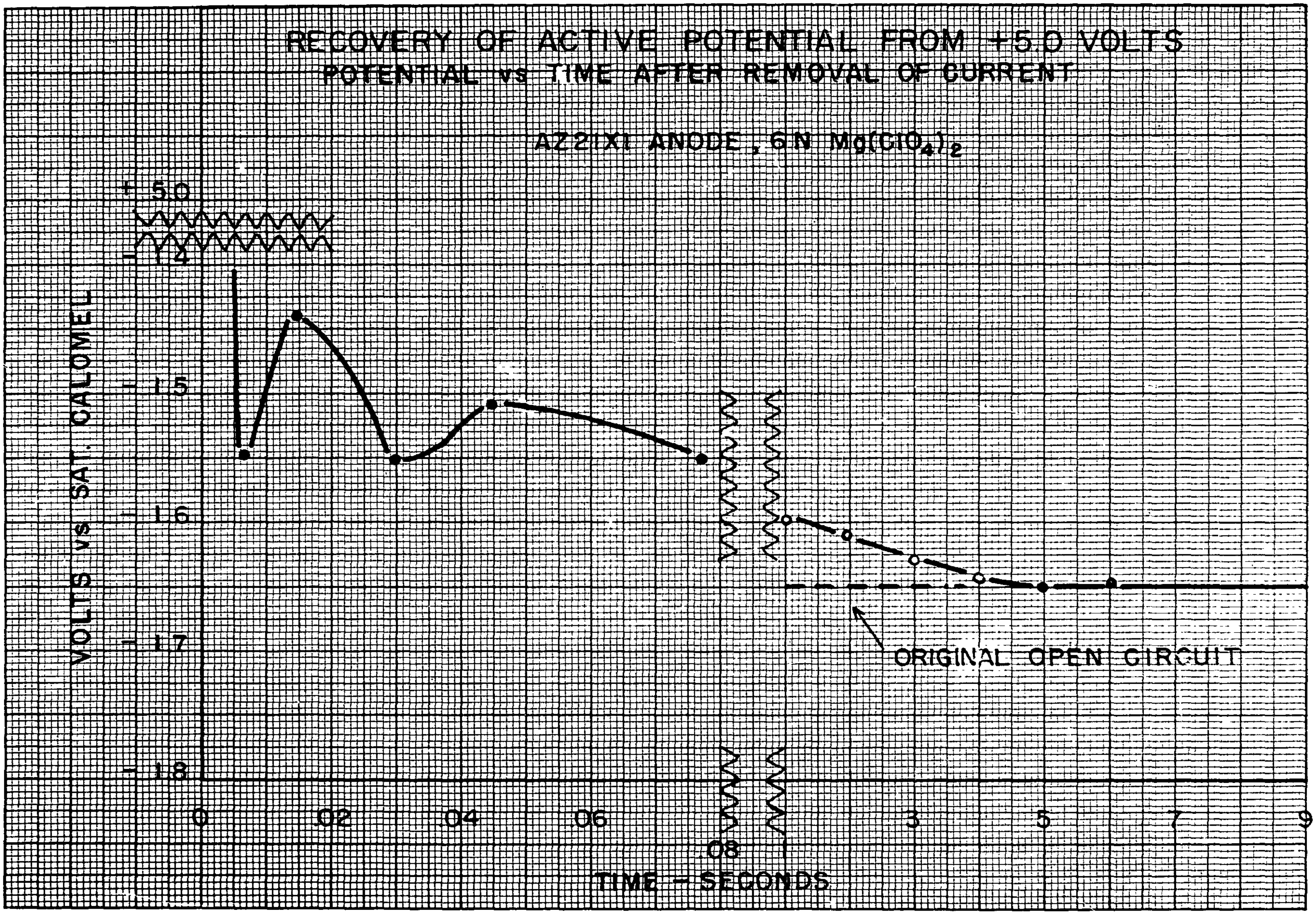
NEGATIVE No. 54497 - 20X

FIG. 13



TIME - SECONDS

FIG. 14



RECOVERY OF ACTIVE POTENTIAL FROM +5.0 VOLTS
POTENTIAL vs TIME AFTER REMOVAL OF CURRENT
AZ21X1 ANODE, 6N Mg(ClO4)2
VOLTS vs SAT. CALOMEL
+5.0
-1.4
-1.5
-1.6
-1.7
-1.8
0
.02
.04
.06
.08
1
3
5
7
9
ORIGINAL OPEN CIRCUIT
TIME - SECONDS

FIG. 15

INDEX CARD

AD__________ Accession No.__________ Unclassified

Dow Metal Products Company Div.,
The Dow Chemical Company

Investigation of the Magnesium Anode
by J. L. Robinson

Second Quarterly Progress Report,
1 October 1962 to 1 January 1963
Illustrations - Graphs, 44 pp
Signal Corps Contract DA36-039-SC-89082
DA Proj. No. 3A99-09-001-02
Unclassified Report

The anodic efficiency and potential behaviors of magnesium were investigated. Mix acetate-perchlorate electrolytes improved the low drain performance of magnesium dry cells.

1. Primary Cells
2. Magnesium Dry Cells

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2. Magnesium Dry Cells

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1. Primary Cells
2. Magnesium Dry Cells

AD__________ Accession No.__________ Unclassified

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1. Primary Cells
2. Magnesium Dry Cells

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